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Post-mortem study of magnesia-chromite refractory used in Peirce-Smith Converter for copper-making process, supported by thermochemical calculations

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ABSTRACT

A cost driver in copper production is the periodical relining of the furnaces because of wear. This paper presents a post-mortem study carried out in a Peirce-Smith Converter (PSC). Samples taken from different locations after a 250-batch campaign at Atlantic Copper (Spain) were analyzed.

New phases are generated as a result of the chemical interaction between the molten phases of the bath and the refractory, creating a reaction layer on the hot face of the refractory. This layer has properties that differ from the refractory, so spallation occurs due to mechanical and thermal shocks during operation.

Thermochemical calculations were carried out to support the post-mortem study. An evaluation was made of the chemical interaction between the molten phases and the refractory under different conditions (slag-refractory volume ratio and Fe/SiO₂ ratio in slag). The solidus and liquidus of the new phases, the amount and composition of the spinel and olivine formed, and the density increase of the refractory in the reaction layer were also determined.

1. Introduction

Copper is produced by pyrometallurgy (82%) and hydrometallurgy (18%) [1]. In hydrometallurgy, copper is extracted from low-grade oxide ores through solvent extraction and electrowinning. In pyrometallurgy, copper is refined from polymetallic sulphides in four steps [1–5]: smelting, conversion, fire refining and electrolytic refining. Smelting (continuous) and conversion (batch) are oxidative processes that produce two immiscible liquids, which are separated by decantation: the slag phase that contains the iron as Fe₂SiO₄ and Fe₃O₄; the copper-enriched phase (in smelting matte with 50–70 wt% Cu and in conversion blister copper with 99–99.5 wt% Cu). Off-gas is also produced (30–34 wt% SO₂). Since reactions are exothermic, the process is auto-thermal (1250–1300 °C). Blister copper is fire-refined by injecting air into the bath to remove the residual S (ppm), and the resultant copper is cast to produce anodes (99.5 wt% Cu). Electrolytic refining (batch) transforms the anodic copper into cathode copper (99.99 wt% Cu).

This study focuses on the refractory used in the Peirce Smith Converter (PSC), which is the most widely used in the copper industry [2,6–8]. The PSC was patented in the United States in 1909, and is still the reference for the converting process. The modern PSC (Fig. 1) is

commonly 4 m in diameter and 10 m long, and rotates along its longitudinal axis to allow the molten phases to be poured in and out. The air for oxidation reactions is introduced via 40–55 tuyeres submerged in the molten bath (35–50 × 10³ Nm³/h) at ambient temperature [8].

Improvements in the refractory industry focus on increasing resistance to chemical attack (mainly from slags), abrasion resulting from moving the melted bath inside, and thermal and mechanical shocks [6,9,10]. Nowadays, magnesia-chromite refractory is widely used in the copper industry due to the capacity of its properties to counter the degradation mechanisms that cause the refractory wear [3,6,7,9,11–13].

Despite resistance to chemical attack from the molten phases, this is still the main cause of lining degradation in the furnaces. The chemical interaction between the refractory and the molten phases, or the off-gas, results in the formation of new phases on the hot face of the brick (magnesian-wüstite, forsteritic olivine and new spinels, among others). As a result, the refractory is damaged due to the changes that occur in its properties, volume expansion and internal crack formation. Additionally, thermal and mechanical stress further damage the lining provoked by the chemical attack because internal micro-cracks widen and spalling appears, thus causing the refractory wear [6,7,9,11,13–18].

The chemistry of the refractory is a key influence on performance, so the quality of the raw materials or the production process must be

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taken into account [4]. The raw materials used are: dead-burned magnesia, chromite ore (Chr), electrofused magnesia and electrofused magnesia-chromite (EMC). Magnesia-chromite refractory can be made by applying different techniques, in particular: direct bonded, re-bonded and fused cast. Direct bonded refractories are the most commonly used due to their performance-cost ratio.

Some studies point to porosity as a key factor in the chemical attack process. Pores are the means of transporting the species from the bath to the refractory in order to achieve the reaction between them. Porosity calculations and simulations using finite element models (FEM) have been carried out in order to estimate the degree of infiltration [15].

Chemical studies demonstrate that copper phases have very low wear capacity, and that the slag phase has the most chemical interaction with the refractory resulting in greater wear capacity [6,9,19–21]. Given the slags' wear capacity on the refractory, slag characterization is important, and studies have been carried out to understand their nature and capacity to attack the refractory. The slags' mainly acid character and low viscosity increase wear capacity [6,22]. Laboratory analyses and thermodynamic models have been made using packages such as FactSage® software to evaluate different conditions (composition, the molten phases in contact with the refractory, the oxygen partial pressure and temperature) [6,23–29].

In recent years, multiphysics modeling has been used to predict wear under different operating conditions, due to its low cost and risk; for example, in the study of bath movement in furnaces as the key factor in erosion, or in the study of thermal shocks to predict crack formation and spalling in the refractory [15,30]. Some authors have developed wear models (nodal wear models; NWM) which take into account the new geometry in the transition zone between the bath and the refractory due to wear [31–36].

Real analysis can be carried out by laboratory tests and post-mortem analysis. In the laboratory, industrial or synthetic molten phases can be used [37,38] and tests can be static or in motion [20,39–42]. The latter are more expensive but closer to reality because they take into account bath movement. Datasheets contain information on their properties but this analysis prior to laboratory testing and post-mortem studies is worthwhile [6,9,20,25,43].

Industrial post-mortem analysis after a real working period [2,6,9] provides the most valuable information, but the main challenge is access to industrial furnaces to gather samples. The type of furnace, the position of the refractory in the furnace, working conditions and type of molten phases must all be considered.

Refractory degradation has been studied in different furnaces and processes: secondary copper smelter linings and slags with PbO and Cu₂O [25], pilot-scale induction furnace and rotary kiln using fayalitic slags [18], anode furnaces [9,19,43–45], rotary furnace of secondary lead smelting [46], continuous copper converting with calcium ferrite slag [47], reverberatory furnaces [48], copper-nickel converters [10], submerged arc furnaces [19], among others.

This work focuses on the chemical degradation of the magnesia-chromite refractory used in a PSC at copper making industry. The location of the refractory in the PSC has been taken into account in order to evaluate the refractory degradation as a function of the zone. In addition, the interaction between the slags and the refractory has been modeled using thermodynamic databases to support the post-mortem analysis and to study other scenarios for different situations of composition and the amount of slag in contact with the refractory.

2. Materials and methods

2.1. Sampling and sample preparation

A PSC was sampled after a working campaign of 250 batches at the Atlantic Copper Smelter. The samples were taken from all areas of the PSC in order to evaluate the chemical interaction of the different molten

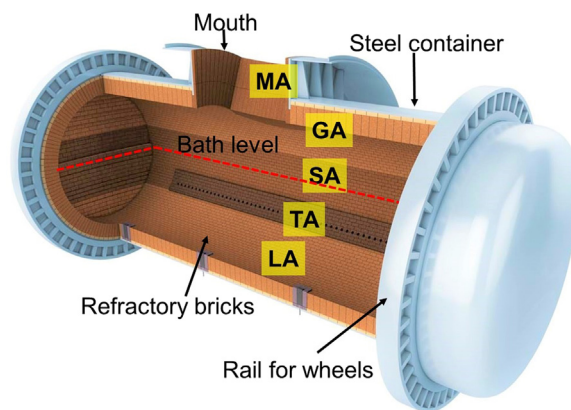


Fig. 1. Drawing of a PSC, which shows the refractory lining, main components, bath level (dotted line) and location of the samples taken for this study (MA, GA, SA, LA and TA). Courtesy of RHI.

phases in contact with the refractory, according to location within the furnace. Fig. 1 shows the converter areas sampled:

- Mouth area (MA): in contact with off-gas and slag.
- Gas area (GA): in contact with off-gas.
- Splashing area (SA): in contact with slag (below the tuyeres).
- Low bath area (LA): in contact with matte and blister copper.
- Tuyeres area (TA): in contact with slag, matte and blister copper.

A central slide (perpendicular to the hot face) was cut from each refractory brick removed to obtain the final samples for analysis from the brick (two or three samples depending on the remaining length of the brick studied). Each sample was divided in two parts: one for FRX/ICP analysis, the other for the SEM/EPMA analysis.

2.2. Analytical methods

Chemical analyses and microstructural characterizations of the samples were performed using a Scanning Electron Microscope (SEM) FEI-QUANTA 200 at the University of Huelva. The SEM is equipped with an Energy Dispersive Spectroscopy (ESEM-EDS) system. The accelerating voltage is 0.2–30 kV with a resolution of 3.5 nm. In addition, it has a secondary Everhardt Thornley detector (ETD) to enable viewing of the surface image, a solid state backscattered electron detector (SSD) to determine the compositional difference and an X-ray dispersion digital microanalysis system (EDAX Genesis 2000) with SiLi detector for quantitative analysis.

Additionally, thermochemical calculations were carried out using thermodynamic databases from the FactSage® software package [26,27,29]. Previously, the mass and energy balance was calculated using the initial matte composition (real one-month average analysis from the Atlantic Copper smelter). Taking this balance and applying FactSage®, a calculation was made of the evolution of the oxygen partial pressure of the system during the slag blow (2.38E-7 atm. at the end) and the composition of the slag produced: 28.47 wt% SiO₂, 53.59 wt% FeO, 12.72 wt% Fe₂O₃, 5.10 wt% Cu₂O, 0.059 wt% FeS, 0.005 wt% Cu₂S. The experiments were designed based on the Fe/SiO₂ ratios for the slag (the main chemical parameter of the fayalitic slags) and the slag-refractory volume ratio (from 0–1 to 1–1). The base volume was the maximum volume of brick affected by the slags, according to the results from the BSE images of the samples.

3. Results

The abbreviation of the species are presented according to the international standard [49].

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